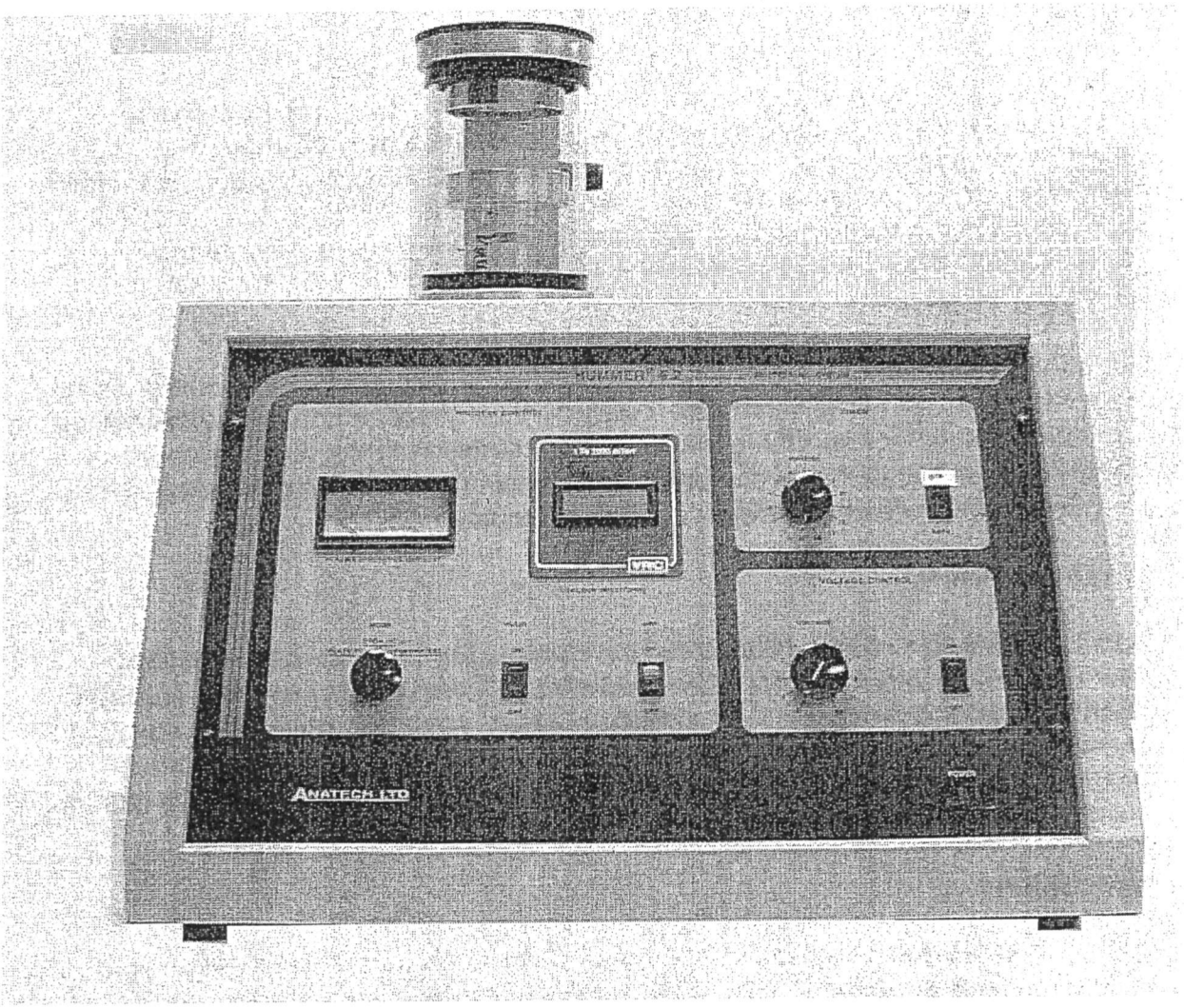
Hummer ™ 6.2
User's Manual



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1 Sputter coating: a brief technical background

Before you unpack, set up and operate your Hummer® 6.2, it is useful to know about sputter coating and how your new instrument works. It isn't absolutely necessary that you read this chapter. However, the information here will help you understand your Hummer and operate it better.

Introduction

In electron microscopy flooding electrons over a specimen produces an image. Typically, specimens examined in an electron microscope are poor electrical conductors and will accumulate a negative charge from the electron flood. This charge distorts the image. To minimize distortion and produce the best images, specimens should have a conductive surface. Even samples that are somewhat conductive will produce a better image if their conductive will produce a better image if their conductivity is increased.

Coating the specimen with metal, usually a precious metal such as gold, palladium or an alloy of the two can increase surface conductivity. If applied correctly, the coating won't impair resolution or surface detail. Several coating techniques have been developed and are routinely used in preparing samples for electron microscopy. Two common techniques are evaporative coating and sputter coating.

Evaporative coating

Evaporative coating involves sublimation of a metal at high temperature and high vacuum. The metal vapor "sprays" the specimen, adhering to any exposed surfaces. This technique is directional, and an irregular-shaped specimen may need to be tilted and rotated to be totally covered. Since the metal is hot, some specimens can be damaged. Further, since metal particle size and coating thickness are hard to control, the results heavily depend on operator skill, technique and care.

Sputter coating

Sputter coating, used in Anatech Hummers, is a cold process in which ions impacting a metal source dislodge metal atoms. The atoms disperse throughout the process chamber in a way that adequately coats irregular-shaped specimens without tilting or rotating them. Since the atoms are cool, there is no thermal damage.

In contrast to the "spray" of relatively large clumps of metal in the evaporative process, sputtering is microscopic process that clouds of metal atoms. This allows the uniformity and thickness of sputter coatings to be easily controlled. In general, sputtering produces high quality and repeatable coatings compared to the evaporative process.

Sputter coating provides two additional benefits. First, sputtering is done in a "soft"

vacuum of 50 to 70 millitorr. Such pressure can be obtained using small, reliable and inexpensive mechanical pumps, in contrast to the costly and elaborate high vacuum pump systems required for evaporative coating. Second, sputter coating efficiently uses material to achieve a desired thickness. Sputter coating is much more efficient than evaporative coating--and results in a lower annual precious metal cost.

Hummer fundamentals

Hummer products operate in three modes useful for preparing specimens: Plate, Etch and Plasma. These have a common element-- the creation and maintenance of plasma. The following sections describe this phenomenon and how it is used in each processing mode.

Plasma production and use

The nature of gas plasmas is described in detail in numerous references (see bibliography at the end of this chapter). The discussion presented here avoids unneeded scientific detail, but provides ample information for working with plasmas and Anatech equipment.

Gas plasma may form when gas is exposed to an electric field. If the field is sufficiently strong, most gas atoms will surrender an electron or two and become ionized. The ionized gas and liberated energetic electrons comprise the *gas plasma*, or *plasma*. Typically, a noble gas in a partial vacuum is used to create plasma. The gas is ionized in the field produced by a hazardously high voltage.

Ionized gas atoms are heavy, but initially have kinetic energy. However, their kinetic energy builds as they accelerate through the field. With high kinetic energy, some of the ions will smash into a negative-charged metal surface, or *sputter source*, and dislodge metals atoms. These metal atoms can float around and will eventually adhere to a specimen.

While ions move to the source, energetic electrons move in the opposite direction. Unfortunately, the latter can hit the specimen and cause it to heat. Biological specimens, polymers or any specimens that are heat-sensitive can be affected and distorted by this heating, leading to artifacts when observed in an electron microscope. Anatech Hummers alleviate electron-heating problems by using a magnet located within the source. Electrons moving toward the specimen will be diverted away from it by the magnetic field.

To further control electrons, a *dark space shield* surrounds the specimen stage. This grounded shield attracts electrons, which may have escaped the effect of the magnetic field. The shield and magnet work together to keep the specimen and stage cool.

Sputtering describes the process of dislodging material from the metal source. *Sputtering rate* specifies the amount of source material removed per unit time. *Sputter*

coating rate, usually expressed as angstroms per minute, is the rate at which a specimen is covered by sputtered material. When the sputtering rate increases, and then the sputter-coating rate increases, since there will be more atoms of souring material floating around.

Many different gases can be used for sputtering. Argon is typically used because it promotes a higher sputtering rate and has a reasonable price. Nitrogen is used less often because it produces a lower sputtering rate and thus a lower sputter-coating rate. Nitrogen gas produces a sputter coating rate half that of argon. Anatech Hummers can only be used with inert gases.

Plate Mode-to plate a specimen with a metal

Anatech Hummers produce plasma for plating in a chamber with a negative-charged source at the top and a positive-charged stage (on which the specimen sits) at the bottom. During Plate Mode, the chamber is evacuated by a vacuum pump, and the operating gas enters through an adjustable leak valve. The plasma forms when a high voltage is applied between the source and stage. Ions accelerate toward the source with energy; proportional to the voltage applied. In general, higher voltage will produce more ions, which increases the sputter rate and the current.

Etch Mode-to remove material from a specimen

In Etch Mode, the polarity of the stage and target are reversed. During Etch Mode, ions will dislodge material from the specimen's surface (similar to metal atoms being dislodged from the sputter source during Plate Mode). Material is etched from the specimen substantially slower than the sputtering rate achieved in Plate Mode. Additionally, Etch Mode causes considerable heating of the specimen.

If etching continues too long (over two minutes), the source can be contaminated by material "back-sputtered" from the specimen. **To avoid contaminating a precious metal source during prolonged etching, replace it with the aluminum etch ring.**

Also, if your Hummer is equipped with the Digital Thickness Monitor (DTM), **always remove the DTM crystal before using Etch Mode**. Handle the DTM crystal carefully—don't get fingerprints on the crystal or debris inside the crystal can.

Plasma Mode--to clean a specimen

Plasma processing is used for ashing, cleaning and etching of metals, ceramics, glasses, organics and composite materials. In contrast to Plate and Etch Modes, which use direct current, Plasma Mode uses alternating current. The resulting plasma is sufficiently energetic to remove small amounts of organic or volatile contaminants from specimen surfaces, and for cross-linking of organic specimens.

An exposure of about two minutes in Plasma Mode before plating improves scanning

electron microscope images. In particular, this treatment helps reduce accumulation of contamination caused by the electron beam. Transmission electron microscope grids processed in Plasma Mode are more hydrophilic.
If plasma processing continues too long (over two minutes), the source can be contaminated by material "back-sputtered" from the specimen. **To avoid contaminating a precious metal source during prolonged etching, replace it with the aluminum etch ring.**

Also, if your Hummer is equipped with the Digital Thickness Monitor (DTM), **always remove the DTM crystal before using Etch Mode**. Handle the DTM crystal carefully—don't get fingerprints on the crystal or debris inside the crystal can.

Pulse Mode--to improve process uniformity and reduce specimen heating

Any of the three Hummer modes described above can be operated in a repeated on-off-on-off pattern, called Pulse Mode. During continuous sputter coating, deposited material tends to aggregate in discrete areas, called "islands". Pulse Mode helps reduce island size. Also, Pulse Mode helps minimize specimen heating. With minimal heating, however, overall process time increases for a given coating thickness.

Hummer high voltage safety

All Hummers are equipped with interlocks or software safeguards to prevent high voltage from being generated unless the top plate is closed and the chamber is under vacuum. The top plate interlock is mounted near the hinge. The vacuum interlock is set so the high voltage is disabled when the chamber is vented and is enabled when the chamber is evacuated. **These interlocks are important safety features—don't override them.**

Bibliography

Thin Film Processes, edited by John L. Vossen and Werner Kern. Academic Press, New York, 1978.

Ion Bombardment of Solids, by G. Carter and J. S. Colligon. Am. Elsevier, New York, 1968.

Atomic and Ionic Impact Phenomena on Metal Surfaces, by M. Kaminsky. Academic Press, New York, 1965.

Handbook of the Thin Film Technology, edited by L. I. Maissel and R. Glang. McGraw-Hill, New York, 1970. See articles by L. I. Maissel and by G. K. Wehner and G. S. Anderson.

Ion Implantation, Sputtering and Their Applications, by P. D.Townsend, J. C. Kelly and N. E. W. Hartley. Academic Press, New York, 1976.

etch or plasma process using the instructions in the next chapter.

If you are not planning to process specimens now, close the main process gas valve at the tank.

3 Operating your Hummer 6.2

This chapter contains the primary operating instructions for the Hummer® 6.2. How you want to process your specimens determines where you need to go in this chapter:

- To plate your specimens, go to the instructions starting on this page.
- To etch your specimens, go to the instructions starting on page 19.
- To plasma your specimens, go to the instructions starting on page 23.

After you become familiar with these processes, you can use the operating summaries in Appendix A.

Getting ready to plate

1. Set Main Power Switch off.
2. Move the top plate to its fully open position. Remove the glass deposition chamber.
3. Install a sputter source (usually gold/palladium or gold):

CAUTION: Always handle the sputter source with lint-free, powder free gloves. Bare fingers will contaminate the sputter source.

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   a. Find the three holes on the back of the sputter source. Line up these holes with the three banana plugs in the top plate.

   b. Position the sputter source over the banana plugs. Gently and firmly press the sputter source into place.

      To remove the sputter source, slide the hooked end of the sputter source extractor between the sputter source and the magnet cover. Rotate the extractor to engage the sputter ring near a banana plug, then pull it out about 1/10 inch (2 mm). Move the extractor to another spot near another banana plug and pull again. Repeat near each banana plug while holding your other hand over the sputter source (to prevent it from falling when it pulls free).

4. Place specimens on the stage. The holes in the stage accommodate standard SEM stubs, which are useful for holding small specimens.

5. Install the glass deposition chamber on the bottom plate.

6. Gently lower the top plate. Listen for the "click" of the high voltage interlock switch. (If the interlock switch is not actuated, plasma cannot be generated.)

7. Open the main process gas valve at the tank. Set the second stage of the gas regulator to 5-6 psi (35,000-41,000 N/m2 or 35-41 kPa).

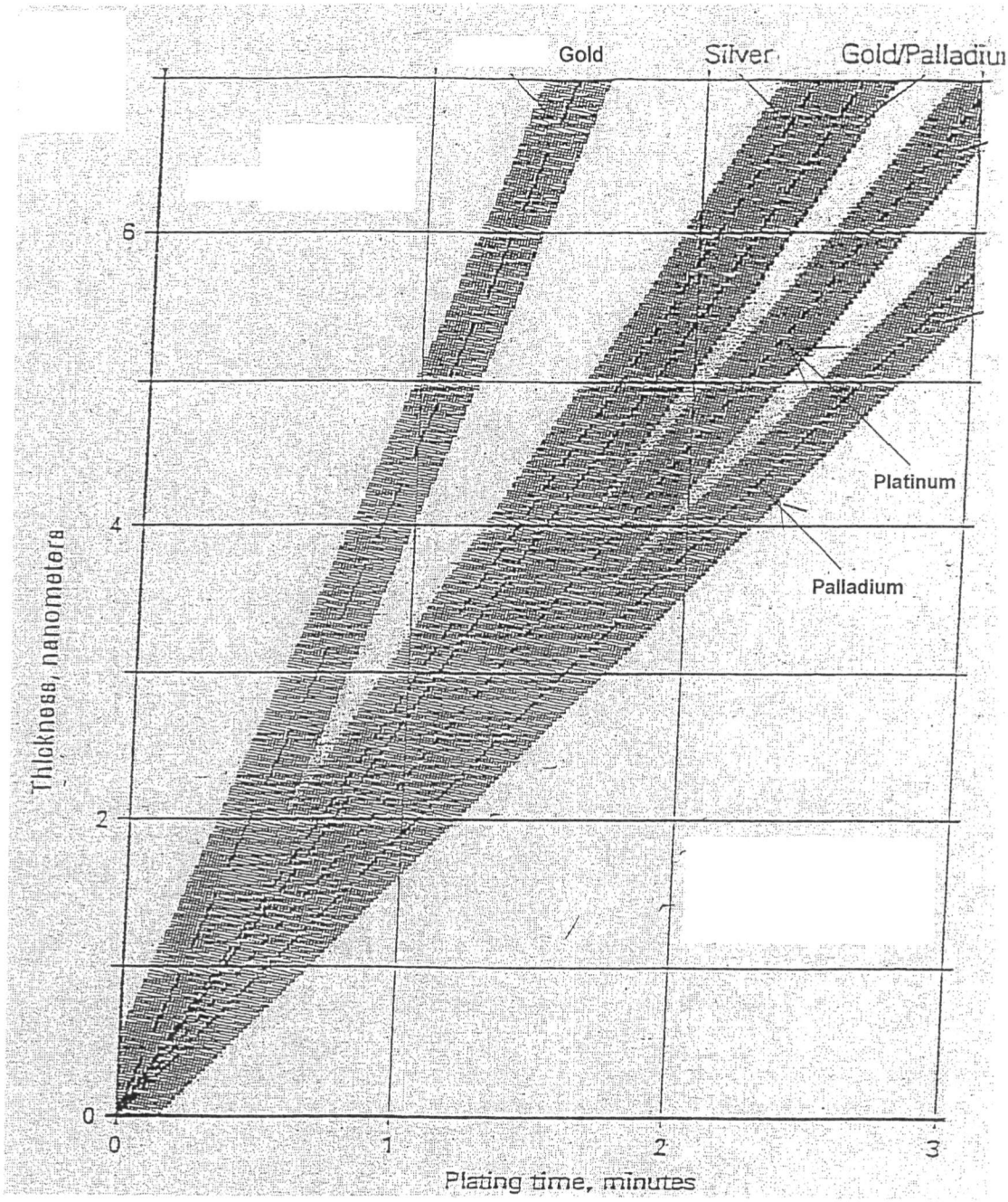
CAUTION: Higher gas pressure can damage the Fine Gas Control Valve and can give the appearance of a leak.

CAUTION: Argon displaces air. Provide adequate ventilation when using the Hummer.

8. Decide on a plating time. Coating thickness depends on plasma voltage and current, vacuum level, process gas, sputter source material and other factors. The time you select is a matter of judgement, based on your experience and evaluation of previous specimens. You are encouraged to experiment to develop a feel for the plating times that are appropriate for your specimens and operating conditions. This set of operating conditions is a good starting point:

Variable	Value
Sputter source material	Gold/palladium or gold
Process gas	Argon
Chamber vacuum	60-80 millitorr
Voltage Control	Position 8.5
Plasma current	15 milliamps
Plating time	2 minutes

Coating rates shown in the graph (on next page) provide some additional guidelines for plating time.



Hummer 6.2 approximate sputter coating rates

Conditions:

Process gas	Argon
Chamber vacuum	70 ± 10 millitorr
Voltage control	Position 8.5
Plasma current	15 milliamps

Rates for your specimens will vary according to the Specific conditions present during each run.

Plating

1. Follow instructions in *Getting ready to plate*, above.

2. Set the operating controls as follows:

Control	Set to
Main Power Switch	Off
Voltage Switch	Off
Voltage Control	0
Gas On/Off Switch	Off
Manual/Auto Switch	Manual
Pulse	Off
Mode Switch	Plate

CAUTION: Do not turn the mode switch while the Voltage Switch is on. Failure to comply could damage the power supply or cause bodily injury.

3. Turn the Main Power Switch on.

1. Flush the chamber with process gas:

   a. Allow the vacuum to reach 30 millitorr.

   b. Turn the Gas On/Off Switch on.

   c. Allow the vacuum to rise to 200 millitorr. Adjust the Fine Gas Control Valve as needed.

   d. Turn the Gas On/Off Switch off.

   e. Allow the vacuum to reach 30 millitorr.

   f. Repeat this steps 4.a. to 4.e. two more times.

5. Turn the Gas On/Off Switch on. Adjust the Fine Gas Control Valve for a vacuum of 40 to 60 millitorr.

6. When the vacuum has stabilized at 40 to 60 millitorr, turn the Voltage Switch on.

7. Turn the Voltage Control clockwise to achieve about 15 milliamps on the Current Meter.

NOTE: When the plasma first appears, chamber vacuum will fluctuate due to molecular adhesion of water vapor, atmospheric gases and solvents. Vacuum will also fluctuate if sputter source is not clean.

NOTE: If the plasma does not appear, make certain the Manual/Auto Switch is in the Manual position.

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8. Alternately adjust the Fine Gas Control Valve and the Voltage Control to achieve stable conditions of 15 milliamps current at 60 to 80 millitorr.

NOTE: When conditions have stabilized, the Voltage Control should read between about 8 and 9 for 15 milliamps of current.

IF

- Voltage Control < 4 and current = 15 milliamps, or
- Voltage Control =8-9 and current > 35 milliamps,

THEN

- Shut down the system (see step 11) and
- Go to Chapter 5, Troubleshooting, and carry out the recommendations for Symptom 3.

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9. **If you want automatic timing:**

   a. Set the Manual/Auto Switch to Auto.

   b. Set the Timer to the desired time. (The plasma will automatically stop after the time you set.)

10. If you want pulse operation, set the Pulse Switch on. (Remember that during pulse mode, the plasma is on only half the time. Adjust your manual or automatic time accordingly.)

11. When desired plating time has been reached, shut down the system:

    a. Set Voltage Control to O.

    b. Turn Voltage Switch off.

    c. Turn Gas On/Off Switch off.
    d. Turn Manual/Auto Switch to Manual (if automatic timing was used).

e. Turn the Pulse Switch off (if Pulse Mode was used).

f. Turn Main Power Switch off.

g. Allow the system to vent to the atmosphere (about 1 to 2 minutes).

12. Open top plate, remove glass chamber and remove specimens.

If you are not planning to process more specimens, close the main process gas valve at the tank.

For abbreviated plating instructions, see Appendix A.

Plating--operating summary

Hummer 6.2

1. Main Power off.
2. Install sputter source.
3. Put specimens on stage.
4. Install glass chamber and lower top plate.
5. Set operating controls:

Control	Set to
Main Power Switch	Off
Voltage Switch	Off
Voltage Control	0
Gas On/Off Switch	Off
Manual/Auto Switch	Manual
Pulse	Off
Mode Switch	Plate

6. Main Power on.
7. Flush chamber with process gas three times.
8. Stabilize vacuum at 40 to 60 millitorr.
9. Gas On/Off Switch on. Voltage Switch on.
10. Adjust Fine Gas Control and Voltage Control for stable conditions of 15 milliamps at 60 to 80 millitorr.
11. For automatic timing: set Timer to desired time and set Manual/Auto Switch to Auto.
12. For pulse operation: set Pulse Switch on.
13. When desired plating time has been reached, shut down system:
    a. Voltage Control to O.
    b. Voltage Switch off.
    c. Gas On/Off Switch off.
    d. Manual/Auto Switch to Manual.
    e. Pulse Switch off.
    f. Main Power off.
    g. Allow system to vent to atmosphere.
14. Remove specimens.

After processing all your specimens:

- Turn off process gas main valve at tank.
- Clean deposition chamber components.

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